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Destruction of PCB-Contaminated Soils with a High-Temperature Fluid-Wall (HTFW) Reactor

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DESTRUCTION OF PCB-CONTAMINATED SOILS WITH A HIGH-TEMPERATURE FLUID-WALL (HTFW) REACTOR

by

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Contract Number 68-03-3014

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16. ABSTRACT

The objective of this project is to demonstrate the feasibility of a high temperature fluid-wall (HTFW) Reactor to detoxify biorefractory hazardous substances, as well as soils contaminated with hazardous materials, such as PCBs, dioxins, and organophosphates. A typical HTFW Reactor consists of a vertical, porous carbon cylinder surrounded by six carbon or silicon carbide electrically conductive rods that heat the core to ca. 2200°C. The core and heating elements are surrounded by a heat shield that is fabricated principally from graphite and forms a "blackbody" chamber. Nitrogen or argon, which is forced into the heating chamber, passes through the porous core to form a protective gas blanket over the inner surface and thus isolates the core wall from attack or accumulation of the hazardous substances or their reaction products. Because of the high radiation density at the operating temperature (2500°K), energy tranfer is predominantly by absorption of radiation rather than by conduction or convection. The material to be detoxified is absorbed onto fine particles (carbon or clay) that acts as heat absorbs. These contaminated particles are then introduced at the top of the core.

For materials with good thermal absortivity, initial surface heating rates exceed 10^7 °K/sec. As the reacted material passes from the hot zone, the products experience on a equally high rate of cooling. The high equilibrium temperature within the core results in degradation of the hazardous substances to simple atomic or molecular forms: the rapid rate of cooling precludes formation of potentially harmful intermediates or byproducts.

The hot zone of the small research reactor used in this study has a 3-in. diameter core and is 3ft long. In a typical test, fine carbon or soil, spiked with a PCB (1% Aroclor 1242), was dropped through the reactor at 100 g/min for 20 min. Representative solid and vapor samples were collected for analysis. Analyses were performed by gas chromatography (GC) with electron capture detection. The results show an overall reduction in concentration of PCB by factor of 2.3×10^{-6}, corresponding to a destruction and removal efficiency (DRE) of 99.9997%.

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DESTRUCTION OF PCB-CONTAMINATED SOILS WITH A HIGH-TEMPERATURE FLUID-WALL (HTFW) REACTOR[1]

by

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ABSTRACT

The U.S. Environmental Protection Agency (EPA) is expending considerable effort in the development of mobile treatment systems for cleanup of releases of hazardous substances from accidental spills or at uncontrolled waste sites. The objective of one such project is to demonstrate the feasibility of a high temperature fluid-wall (HTFW) Reactor to detoxify biorefractory hazardous substances, as well as soils contaminated with hazardous materials, such as PCBs, dioxins, and organophosphates.

A typical HTFW Reactor consists of a vertical, porous carbon cylinder surrounded by six carbon electrically conductive rods that heat the core to ca. 2200^{o}C. The core and heating elements are surrounded by a heat shield that is fabricated principally from graphite and forms a "blackbody" chamber. Nitrogen or argon, which is forced into the heating chamber, passes through the porous core to form a protective gas blanket over the inner surface and thus isolates the core wall from attack or accumulation of the hazardous substances or their reaction products. Because of the high radiation density at the operating temperature (2500^{o}K), energy tranfer is predominantly by absorption of radiation rather than by conduction or convection.

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1. Work performed by Baird Corporation as a subcontract under EPA contract No. 68-03-3014 with Rockwell International Corp. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the U.S. EPA.

The material to be detoxified is adsorbed onto fine particles (carbon or clay) that act as heat absorbers. These contaminated particles are then introduced at the top of the core.

For materials with good thermal absortivity in the near infrared, initial surface heating rates exceed 10^7 ^oK/sec. As the reacted material passes from the hot zone, the products experience an equally high rate of cooling. The high equilibrium temperature within the core results in degradation of the hazardous substances to simple atomic or molecular forms; the rapid rate of cooling precludes formation of potentially harmful intermediates or byproducts; the very rapid rate of heating permits a high throughput rate even though the residence time in the HTFW Reactor is short.

The hot zone of the small research reactor used in this study has a 3-in. diameter core and is 3 ft long. In a typical test, fine carbon or soil, spiked with a PCB (1% Aroclor 1242), was dropped through the reactor at 100 g/min for 20 min. Representative solid and vapor samples were collected for analysis. The vapors sampled were trapped by a polyurethane plug in a glass cartridge. Analyses were performed by gas chromatography (GC) with electron capture detection.

The results show an overall reduction in concentration of PCB by a factor of 2.3×10^{-6} with a standard deviation of 0.8×10^{-6}, corresponding to a destruction and removal efficiency (DRE) of 99.9997%. No hazardous pyrolysis products were observed using GC with electron capture detection.

INTRODUCTION

The use of high temperatures to disintegrate organic chemical substances or to incorporate inorganic toxic wastes into glasses has been practiced, for many years; however, conventional process equipment operating much above 1200^oC has generally encountered limitations in materials of construction.

Most refractory liners either degrade mechanically or dissolve in the slags found in many waste streams. Maintenance of pyrolysis or incineration equipment with moving parts in the high temperature region presents a potentially severe problem when uncharacterized feed materials with fusible species are introduced or precipitable products are formed.

The High Temperature Fluid Wall (HTFW) Reactor

A typical HTFW Reactor consists of a porous carbon core surrounded by six carbon electrical heaters that heat the core to approximately 2200°C. The core and electrodes are enclosed by the heat shield made principally of graphite, which forms a blackbody cavity. Nitrogen, which surrounds the heaters, is forced through the porous core to form a protective gas blanket over its inner surface and to protect the core from the reactants and their products. A partial vertical cross section of a typical Thagard Reactor is shown in Figure 1, and a horizontal cross section is shown in Figure 2.

The blackbody power radiated by any substance at a temperature T is given by $P = \sigma \varepsilon T^4$ where:

P = Power radiated per unit area, W cm^{-2}

σ = Stefan-Boltzman constant, 5.7 $x10^{-12}$ W $cm^{-2}T^{-4}$

ε = emissivity of the substance, 0 1

T = absolute temperature of the substance, °K

For T = 2500°K and an emissivity of unity the power density will be greater than 200 W/cm^2, with radiation peaked at approximately 1.15u in the near infrared. In this case energy transfer to reactants is primarily by absorption of radiation rather than by conduction or convection. For a good absorber such as carbon, initial surface heating rates will be in excess of 10^7 degrees/sec and process temperature will be reached in a small fraction of a second. As the sample leaves the hot zone and "sees" cold walls the reverse energy transfer occurs and the sample experiences similarly high initial rate of cooling.

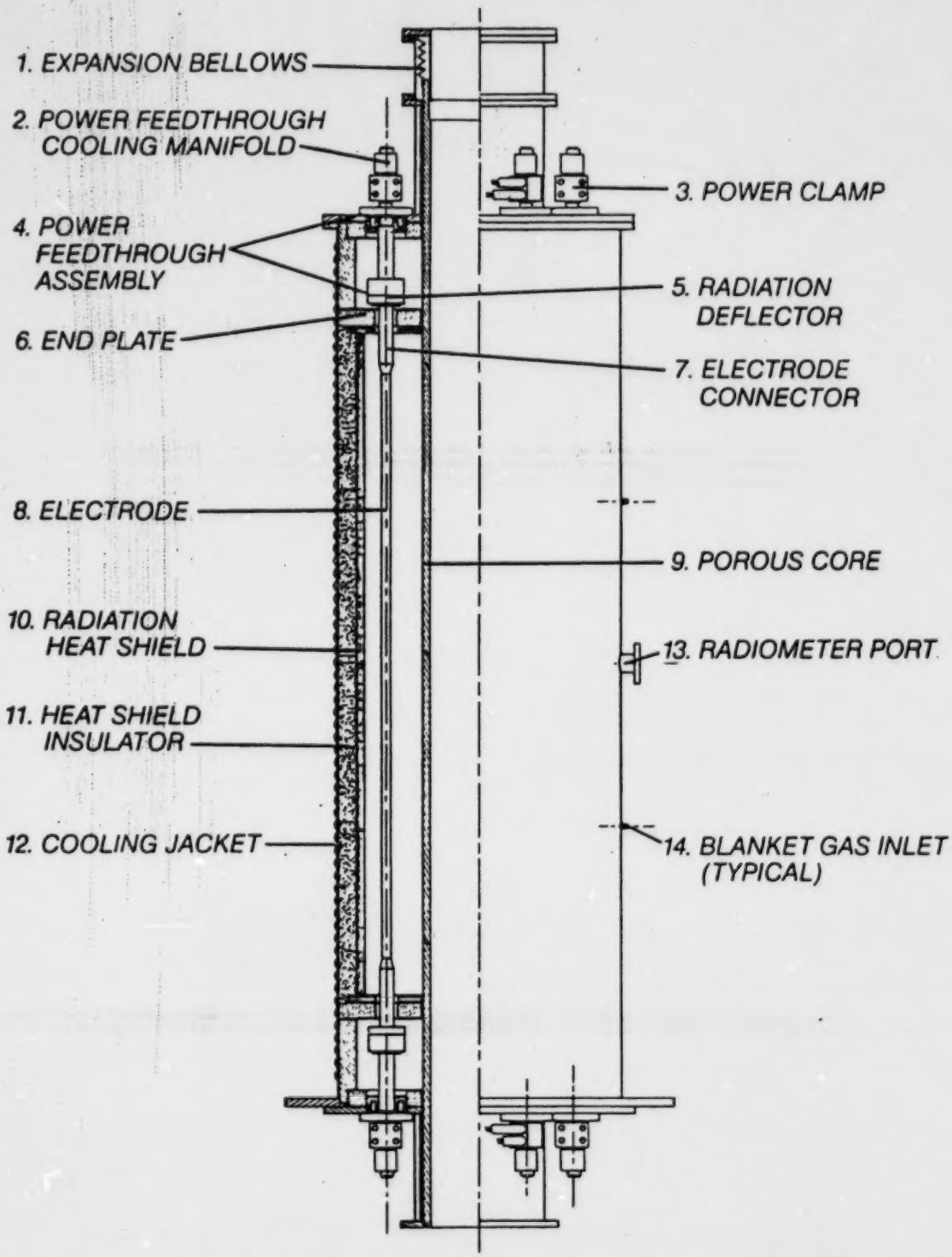

Figure 1. Vertical cross section of a typical Fluid-Wall Reactor

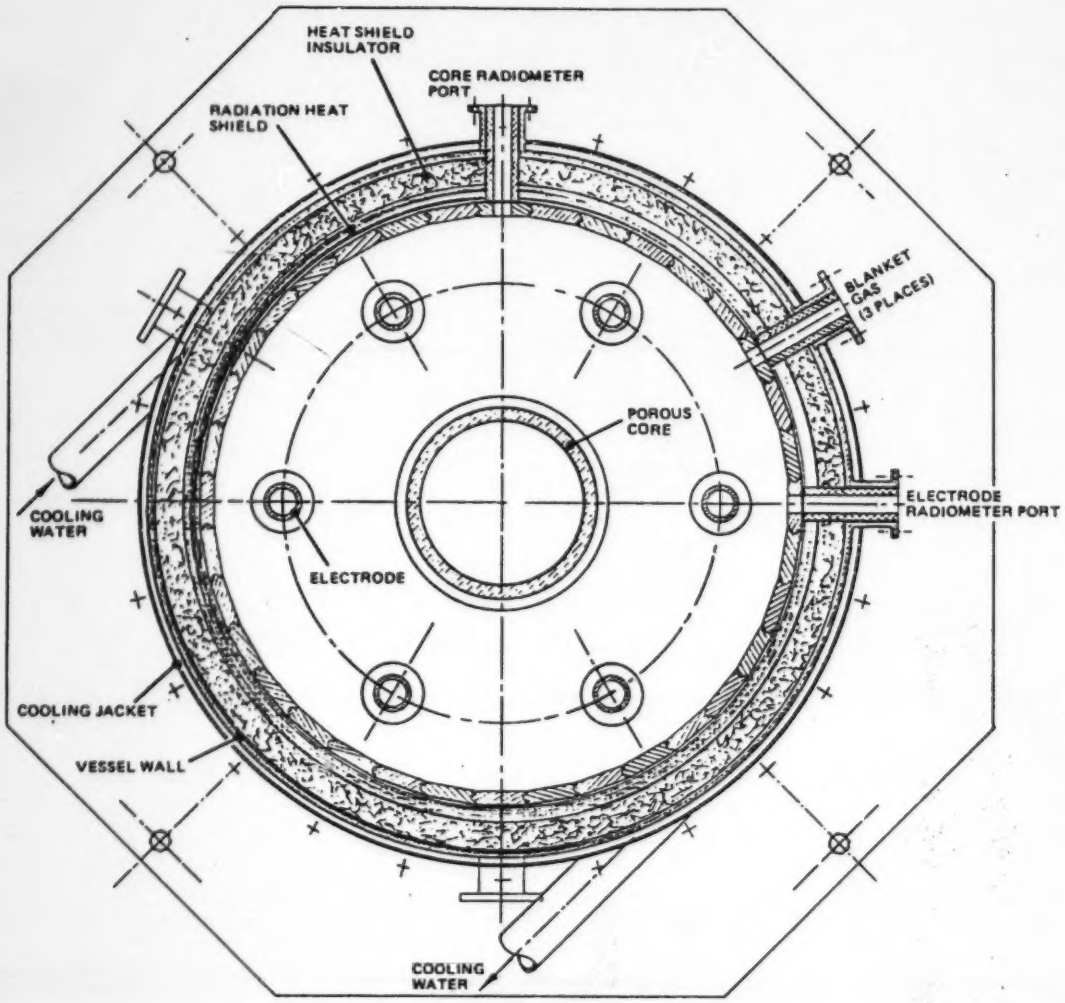

Figure 2. Horizontal cross section of a typical Fluid-Wall Reator

The high temperature within the core guarantees that samples will be reduced to atomic or very simple molecular forms. The high rate of cooling prevents reformation of potentially hazardous recombination products, and the high rate of heating allows relatively large processing rates at short residence times. Hydrocarbons are readily pyrolyzed to $C+H_2$; oxygen-bearing hydrocarbonaceous materials are gasified to $CO+H_2+C$; halogenated organic compounds are decomposed to $C+CL_2+HCL$. In the last case, the addition of calcium hydroxide results in the "gettering" of the chlorine to form $CaCL_2$. Inorganic compounds such as clay which melt below the Reactor core temperature are fused into vitreous solids. Most metal salts such as $CaCL_2$ are soluble in such molten glasses, leaving vitrified beads with the salts locked up in solid solution.

Typical Operation

Prior to introduction into the HTFW Reactor, feedstock must be reduced to approximately -100 mesh. Referring to the system flow diagram of Figure 3, the feed is placed into the hopper which is then sealed. A calibrated Vibra-Screw feeder carries the feed into the top of the reactor where it is entrained in sweep nitrogen and carried into the reactor, first passing through the mechanical disperser. The function of the vibrating screen disperser is to assure adequate dispersal of the feed without agglomeration. Passing through the reactor proper, the feed undergoes rapid heating and subsequent cooling, during which time hazardous materials are disintegrated and any ash matrix may be vitrified. Large solids fall by gravity into the sealed solids collection chamber (pan), while gasses and entrained fine solids are carried off, first to a cyclone and then to a baghouse for particulate removal. Finally, the effluent vapor passes through a venturi scrubber before being released to the atmosphere.

Effluent Sampling

As indicated in Figure 3, two principal effluent samples are collected for analysis. Gaseous samples (with entrained fine particulates) are withdrawn continuously from a point in the discharge line directly before the cyclone and baghouse, using a calibrated pump. Since the gas flowing through the Reactor is controlled (i.e. the Reactor is a closed system with no leaks), a metered sample flow may be related to total throughput. The vapor samples pass through a water-cooled heat exchanger prior to entering a glass cartridge containing a pre-extracted polyurethane plug. After collection, the cartridge is removed and packaged for shipment to an analytical laboratory. (The glass cartridges containing pre-extracted polyurethane plugs are prepared following the design of Lewis and MacLeod (1982) of EPA-RTP). The cartridge also contains a glass filter ahead of the plug to collect excess particulates. The heat exchanger is a cold trap which can be shipped intact for analysis of trapped materials. Solids fall into a pan at the bottom of the Reactor. A clean metal can is placed into the pan for automatic collection of samples. At the termination of a run, the can is capped immediately prior to shipment for analysis. Several other effluent samples may be collected. For chlorine determination in the gas(as HCl or Cl_2), bubblers sample a metered portion of the output gas, following particulate removal. The solids collected in the cyclone and baghouse may be analyzed separately to relate any detected species to particle size.

EXPERIMENTS

Preparations

The original plans for this program called for natural samples of PCB on sediment, perhaps from an uncontrolled dumpsite. Difficulties in obtaining suitable representative natural samples caused a shift to artificially

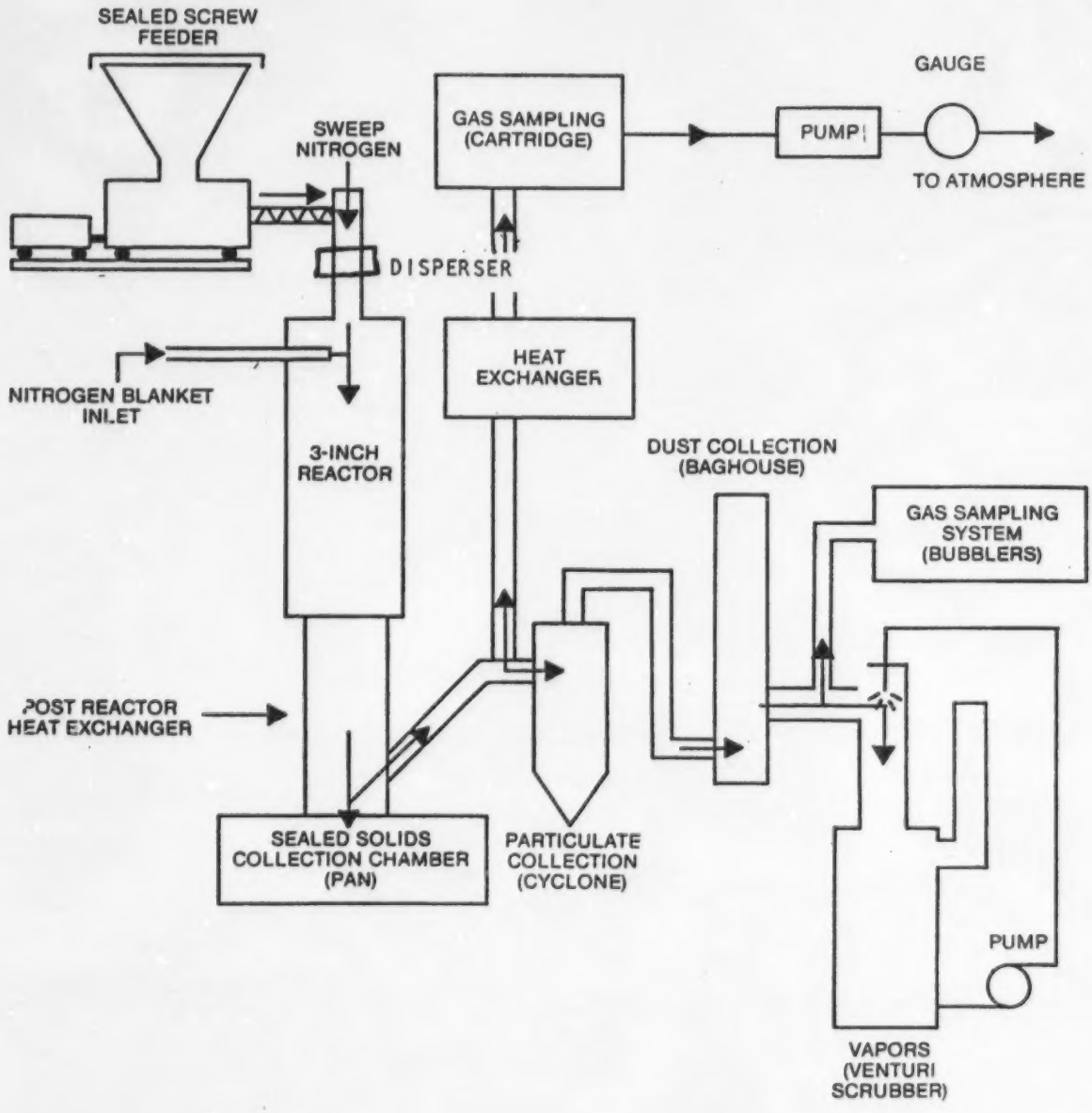

Figure 3. Schematic Diagram of the HTFW Reactor System.

prepared samples using a 1:1 bentonite:kaolin mixture as substrate and Aroclor 1242. An initial set of experiments failed because of poor dispersion of feedstock (resulting in incomplete heating of some agglomerates) and because the molten clay condensed on the reactor walls just below the hot zone. The former problem of poor dispersal was partially ameliorated by increasing the vibrational frequency and amplitude of the disperser located just below the feed hopper (Figure 3). Even with this improvement the bentonite:kaolin mixture showed some evidence of clumping in a cold run. Further, hot runs showed more evidence of condensation on walls. Therefore, the matrix materials was changed to fine carbon (which does not vitrify) and a local soil (which had been used sucessfully before). In further attempt to guarantee complete heating of the samples, the core temperature was increased from 2200°C to approximately 2340°C. The final test matrix is given in Table 1.

TABLE 1. TEST/SAMPLING MATRIX

	Test	Feed	Pan	Cart-ridge	Heat Exchanger
1.	Carbon	X	X	X	
2.	Carbon & PCB	X	X	X	
3.	Carbon		X	X	
4.	Carbon & PCB		X	X	
5.	Carbon		X	X	
6.	Carbon & PCB		X	X	X
7.	Soil	X	X	X	
8.	Soil & PCB	X	X	X	
9.	Soil		X	X	
10.	Soil & PCB		X	X	
11.	Soil		X	X	
12.	Soil & PCB		X	X	X

Feedstock Preparation

Feedstock preparation involves extensive pulverizing and blending of one percent Aroclor 1242 on carbon or soil to form homogeneous -100 mesh samples.

Preliminary Tests

Having completed tests on cold and hot substrates (carbon and soil), which indicated the shaker was dispersing the sample better and the slag appeared to have reached temperature, it was decided to perform two single tests on carbon plus PCB and soil plus PCB, similar to those in the matrix, but without any background runs. The actual matrix of tests would not be continued unless the preliminary tests indicated the reactor was working well. These tests were performed on September 15, 1982 and the reactor seemed to perform well. The only sample analyzed was from the pan since it was felt that it would indicate how well the reactor was working. The results for pan only were that the fractions remaining were 1.46×10^{-6} for the carbon substrate, and 6.22×10^{-6} for the soil substrate. Based on these results, it was decided to carry out the full set of tests.

Typical Test

A feedstock containing one percent Aroclor 1242 was introduced into the reactor at a rate of approximately 100g/min for 20 min. The actual weight introduced was determined by subtracting the weight of material remaining in the hopper from the original weight.

The tests, following the matrix in Table 1, were carried out on October 26-27, 1982.

Effluent Analysis

Rockwell Environmental Monitoring and Service Center (EMSC) was the primary analytical laboratory; approximately one third of the samples analyzed by West Coast Technical Services (WCTS) as a check. It was felt originally that a combination of gas chromatography and mass spectroscopy (GC/MS) would be necessary to eliminate possible interferences such as were encountered in

the earlier hexachlorobenzene (HBC) work. However, on the recommendation of the primary analytical laboratory, GC with electron capture was selected as the primary analytical methodology, following EPA method 601 (In Test Methods for Organic Chemical Analysis of Municipal & Industrial Wastewater, EPA 600/4-82-057, July 1982) with the option of using GC/MS if identification was questionable. This choice retains the desired analytical sensitivity of better than one part in 10^6.

Feedstock -- The selected matrix was extracted with methylene chloride, concentrated, and examined with GC and electron capture detection.

Cartridges -- The screen, filter (with particulates) and polyurethane foam (PUF) plug were removed from the cartridge and extracted with methylene chloride, concentrated, and examined with GC. The inside of the cartridge was rinsed with methylene chloride as part of the sample examined.

Pan -- The pan samples were expected to be on the order of 1500g far too large for ordinary Soxhlet extractors. Therefore, the extraction took place in large flasks with the large amount of solvent concentrated in a Kuderna-Danish apparatus preparatory to analysis as for the cartridges above.

Heat Exchanger -- The entire heat exchanger glassware was submitted for analysis. The interior was flushed with methylene chloride and the washings plus contents extracted as for the cartridge samples.

RESULTS

Data Reduction

A summary of principal data and results is given in Table 2. In order to interpret this data more clearly, the data in the second to the last column of

TABLE 2. REDUCTION OF DATA FOR EXPERIMENTS OF 26-27 OCTOBER 1982

TEST NO.	INPUT	FEED USED (g)	PCB IN (g)	CARTRIDGE PCB (ADJ) (μg)	PAN PCB (μg)	HEAT EXCH. (μg)	TOTAL PCB OUT (μg)	PCB ADJ. FOR 1000g	FRACTION REMAINING X10^6
1	C	1700		401.7	105.0		506.7	298	
2	C+PCB	1223	12.35	229.3	66.5	5.1	300.9	246	24.6
3	C	2253		93.7	60.3		154.0	68.4	
4	C+PCB	1653	16.70	31.0	16.6	6.9	54.5	32.9	3.3
5	C	2182		160.7	91.9		252.6	119.8	
6	C+PCB	1431	14.45	33.6	12.5	6.0	52.0	36.3	3.6
7	Soil	2016		702.	412.2		1114.2	552.7	
8	Soil+PCB	1909	10.91	48.1	7.3	2.2	57.5	30.1	3.0
9	Soil	2069		17.3	2.2		19.5	9.4	
10	Soil+PCB	1939	10.61	25.0	2.7	2.1	29.8	15.4	1.5
11	Soil	1856		57.7	4.1		61.8	33.3	
12	Soil+PCB	1923	10.77	41.9	4.5	2.1	48.6	25.3	2.5

Table 2 were plotted against "Test No." in Figure 4. These results are adjusted for an input of 1000g feedstock and therefore can be compared directly. For insight, the results for odd-numbered tests were connected with straight lines, as were results for even-numbered tests with PCB. The transition between carbon and soil substrate is indicated by dashed lines.

Destruction Efficiency: Carbon Substrate

An examination of the results tabulated in Table 2 and plotted in Figure 3 shows that the fraction remaining in tests where no PCB was added was often greater than in tests where PCB was added. Hence no simple background subtract is possible. Results are calculated directly from the three tests with carbon and PCB (Tests 2,4,and 6). Further examination shows that the results for Test 2 are almost an order of magnitude greater than for the other two, suggesting this datum may be an outlier that should not be considered. Since the very first background test (Test 1) also showed a large background level of PCB, it may be concluded that the reactor was contaminated at the beginning of the tests, perhaps carrying into the second test. Test 2 was plagued with a number of problems, the chief of which was complete loss of disperser action for at least one-half minute. Since proper dispersal of the feedstock is believed to be vital, it is probable that unreacted PCB passed through the reactor during this time. Therefore we regard the data of Test 2 as outliers and the results are not included in our conclusions.

The principal results for PCB on a carbon substrate are:

1. The average fraction remaining for tests 4 and 6 is 3.5×10^{-6} with a standard deviation of 0.2×10^{-6} corresponding to a destruction efficiency of 99.9997%.
2. The PCB observed on background tests (with no PCB in the feedstock) was higher than on those tests with PCB, indicating a carryover of material in the reactor from test to test.

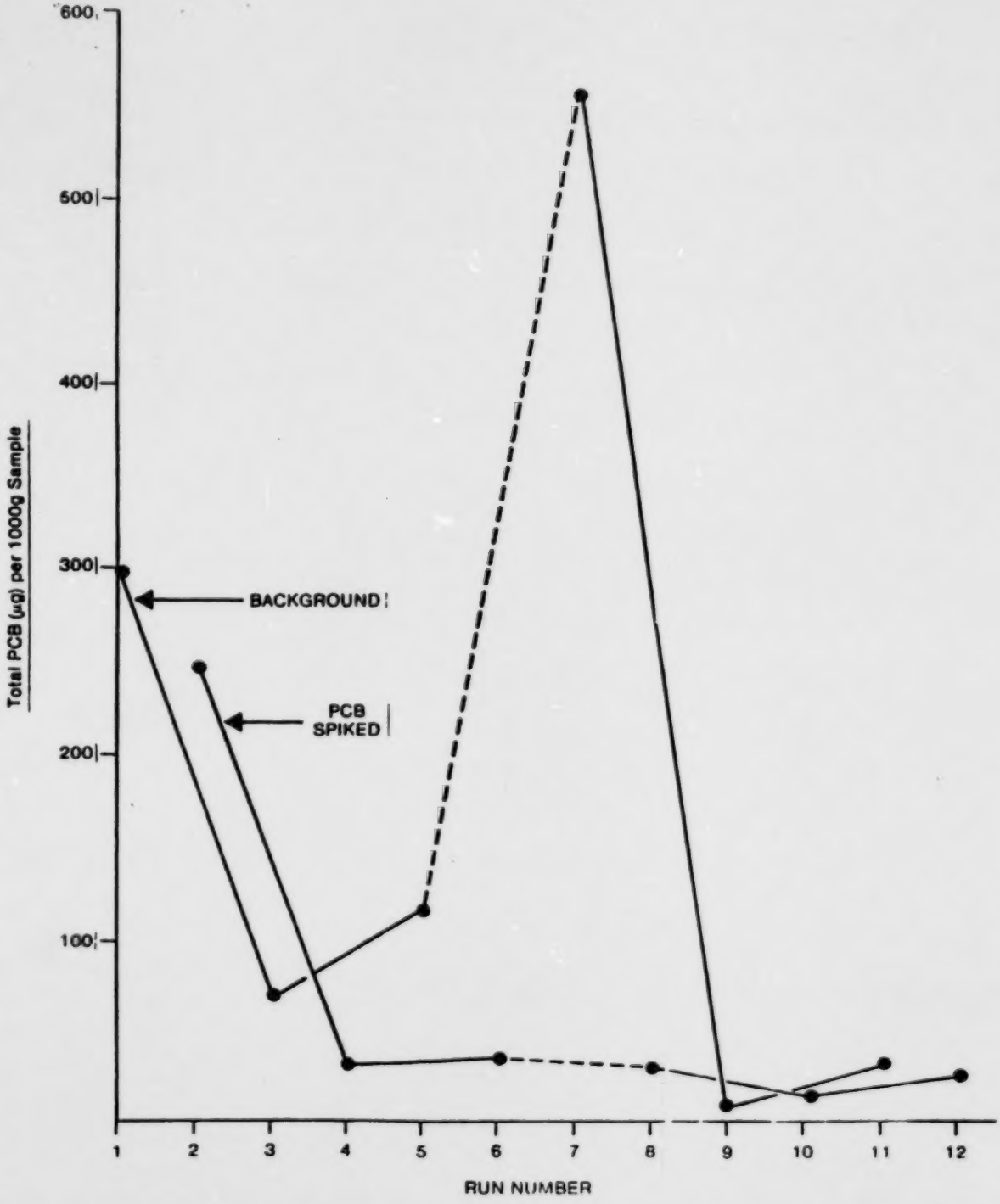

Figure 4. Total PCB (µg) per kilogram of feedstock for all tests; experiments of 26,27 October 1982.

Destruction Efficiency: Soil Substrate

An examination of the results tabulated in Table 2 and plotted in Figure 4 shows that the fraction remaining in tests when no PCB was added was often greater than in tests where PCB was added. Hence, no simple background subtract will be possible and results will be expressed for the three tests with soil and PCB (Tests 8, 10,and 12). Further examination shows that the result for Test 7 is more than an order of magnitude greater than the other two background measurements, suggesting it may be an outlier which should not be considered. This conclusion is supported by a consideration of the problems of Test 7. Back-pressure built up almost forcing cancellation of the run, and there was a leak in the feed system which released some solids.

The principal results for PCB on a soil substrate are:

1. The average fraction remaining for Tests 8, 10 and 12 is 2.3×10^{-6} with a standard deviation of 0.8×10^{-6}, corresponding to a destruction efficiency of 99.9998%.
2. The PCB observed on background tests with no PCB added was often higher than on tests with no PCB added. These anomalous results indicated buildup within the reactor and did not permit background subtract.

Overall Destruction Efficiency

A comparison of results of carbon and soil substrates indicates that the degradation may be slightly more efficient in soil than in carbon; however, results are similar. If all seven PCB tests (omitting the outlier, Test 2) are considered, the overall fraction remaining is 2.8×10^{-6} with a standard deviation of 0.8×10^{-6} corresponding to a degradation efficiency of 99.9997%. These results are summarized in Table 3.

TABLE 3. FRACTION REMAINING & DEGRADATION EFFICIENCY FOR PCB DEGRADATION EXPERIMENTS

	FRACTION REMAINING	STANDARD DEVIATION	DEGRADATION EFFICIENCY
TESTS 4 & 6 (Carbon)	3.5×10^{-6}	0.2×10^{-6}	99.9997%
TESTS 8,10, & 12 (Soil)	2.3×10^{-6}	0.8×10^{-6}	99.9998%
TESTS 4,6,8,10, & 12 (Carbon and Soil)	2.8×10^{-6}	0.8×10^{-6}	99.9997%

Background Anomolies

The anomalous background results suggest that the reactor was not functioning properly. In particular, it is evident that the reactor became contaminated in a manner which coupled experiments--causing PCB to come through when no PCB had been added. This was not noted in earlier measurements on the six-inch reactor and is probably related to the smaller core diameter and the incomplete fluid wall on the three-inch reactor.

Special Results

All tests except Test 11 & 12 were run at a feedrate of approximately 50 g/m. Tests 11 & 12 were run at approximately 100 g/m to ascertain if feedrate had any influence on results at this level. The results for these last two tests were quite similar to earlier results, indicating that feedrates of 50-100 g/m were well below any saturation rate which could cause reduced efficiency. This is in agreement with earlier results.

It was noted that slagging occurred with the soil, and the pan often had large chunks of vitrified material. Therefore Rockwell was requested to analyze the large chunks separately from the fines for Tests 7 & 8 in order to

determine whether observed PCB was associated primarily with either type of material. Test 7 had problems and displayed anomalously high results for both pan and cartridge.

In Test 8 with PCB the observed concentrations were 0.013 g/g and 0.0007 g/g for the fines and chunks respectively. The lower values for the chunks may well be related to the difficulty of analyzing these materials by standard methods. In any case, no particularly meaningful conclusions can be reached about preferential concentration of undestroyed PCBs in fines or aggregates.

CONGLUSIONS

The following conclusions are drawn from experiments using the three-inch research reactor operated at a core temperature of 2343^{o}C at a feedrate of 100 g/min with PCB loading at one percent. Standard tests lasted twenty minutes corresponding to a sample size of approximately 2000g substrate and 20g PCB as Aroclor 1242.

1. An overall reduction in concentration of PCB by a factor of 2.3×10^{-6} was achieved with a standard deviation of 0.8×10^{-6}. This corresponds to a destruction efficiency of 99.9997%, where Percent Destruction Efficiency = (1-total fraction remaining) X 100
2. Destruction was essentially similar for carbon and soil substrates, with slightly higher efficiencies for soil.
3. Destruction efficiency is essentially independent of feedrate in the 50-100 g/m in range for the three-inch reactor at 2343^{o}C.
4. No pyrolysis products other than carbon and hydrogen chloride were observed or identified by either analytical laboratory, using GC with electron capture detection.

5. The method of dispersing feedstock into the three-inch reactor is faulty and needs further development.
6. Slagging, the condensation of liquid or gaseous effluent on the lower cool core surface, was observed with the soil substrate. The cause is believed to be related to the small diameter of the research reactor and particular design which terminates the fluid wall just after the hot zone.

RECOMMENDATIONS

The present experimental program demonstrated a high degree of destruction for PCBs on soil or fine carbon, suggesting the great potential of the High Temperature Fluid Wall (HTFW) Reactor for on-site destruction of such wastes. With support from a private firm Thagard Research has already begun a program to produce a prototype Mobile Reactor by the end of 1983. Vital to the success of this unit will be further understanding of fundamental processes (theoretical and experimental) occuring in the reactor, and improvement in feedstock preparation and dispersion, as outlined in the following recommendations.

Feedstock Preparation and Dispersion

One of the problem areas in the system used in the present program was the irregular and poorly dispersed introduction of solid feedstock. It is recommended that an improved dispersion system be designed and tested with various substrates, especially those with a tendency to clump. The tests should also include an optimization of particle size, with reference to the optical absorption of the feedstock of 2500^{o}K blackbody radiation. Finally, commercial devices for optimum sample preparation should be identified and tested.

Enhancement of Radiative Energy Transfer

Materials such as PCBs are relatively transparent to blackbody radiation at 2500°K. For efficient heating other absorbing materials must be added. (Carbon and soil were used in the present program.) It is recommended that a theoretical and experimental program be carried out to determine optimum materials and particle sizes for radiation absorbers. Also, the best mass ratio of absorber to sample must be established. The study should include optimization of sample preparation procedures (e.g. wet or dry mixing) and include the transfer of energy to vapor samples. Practical equipment should be identified or designed.

Slagging

Factors involved in the condensation of liquid and vapor effluents on cool core walls include core diameter, method of sample introduction, and design of the fluid wall immediately below the hot zone. A theoretical and experimental study should address this important problem, including comparison of slagging in the three-, six-, and twelve-inch reactors available for testing.

Throughput

The throughput employed in this program - 100 g/min - was selected for convenience to demonstrate feasibility. The three-inch reactor was also selected for convenience and availability. It is recommended that an interactive theoretical/experimental program be conducted on a twelve-inch reactor (e.g., the mobile unit now under construction) to determine the basic processes occurring when absorbing and non-absorbing species fall through a blackbody cavity. The goal will be to establish conditions for maximum throughput as a function of the physical characteristics (particle size,

absorbance) of the feedstock. The theory and predictions should be tested for a number of materials.

Hazardous Reaction Products

No toxic or hazardous reaction products were observed during the present program; however, there is a concern that chlorinated dioxins or dibenzofurans may be produced in miniscule but significant quantities. A program designed to search for these materials at very low levels should be carried out to demonstrate that such materials are not generated as reaction byproducts, even at low levels. The program could also verify that NO_x is not generated in the HTFW Reactor.

Economic Study

It is recommended that a preliminary economics study be carried out which will establish probable costs for acquisition, operation, and maintenance of the proposed mobile reactor. The study should include a comparison with rotary kilns and other competitive equipment.